THE PRESERVATION OF CANE JUICES BY REFRIGERATION.

By C. H. KARLSON and W. H. FOSTER.

The Congress continued at 3 p.m.

Before reading his paper Mr. Foster made the following remarks :—

I would like to say a few words before starting this paper. It has become more noticeable in recent years that, as the factory increases and the amount of work in the laboratory increases, there is less time for any real research work for the laboratory staff outside their routine work. Further, we thought if we could have a lot of these samples that we now do hourly, and which are not of immediate importance, done at eight-hourly or even at longer intervals, we could get the same results, probably more accurately, and at the same time liberate more of the chemist's time to deal with other problems that arise from time to time.

One of the most difficult problems confronting the sugar-house chemist is the preservation of his samples. Cane juice in all its stages is an ideal medium for the growth of micro-organisms and usually arrives in the laboratory in a heavily infected state. Since it is neither desirable nor economically possible to analyse accurately every sample as it arrives, recourse is had to various preservatives.

All preservatives used to-day generally lose their influence after a few hours. This means considerable work in the laboratory, and it is not possible to use the most accurate methods. An ideal preservative would be one in which nothing was added to the juice, and which would keep the juice 24 hours or longer without any deterioration. Such a preservative would permit one accurate analysis on a composite, and increased accuracy is highly necessary when one considers that to obtain an increased extraction of one or two points requires the expenditure of thousands of pounds. The basis on which this expenditure is calculated, depends on the chemist and the accuracy of his methods.

Refrigeration offers the ideal preservative. As a method it is not new. Vast industries are based upon its preservative action, but in the past it has been too expensive for use in the sugar house laboratory. The apparatus was bulky and did not lend itself to small scale operation. However, in recent years this has been changed. To-day, in millions of households are found small refrigerators of a variety of makes and these place within the reach of every chemist an ideal preservative of cane juice samples.

The authors of this paper have carried out a series of experiments on preserving sugar mill products by means of refrigeration. We are indebted to the Tongaat Sugar Company for permission to use their laboratories and to the British Electrolux Company for the loan of a refrigerator.

The Electrolux used was the six cubic feet capacity standard household type. It served our purpose admirably, but for regular work the standard ten cubic feet type would probably be better. Quick cooling is essential and we had some trouble in the beginning through overloading the machine. Slow cooling permits some action of the yeasts, especially in highly infected juices, such as secondary juice. We obtained the best results by keeping the temperature at about minus 10 degrees centigrade. At this temperature, the samples froze solid in a few minutes and in that state will keep indefinitely. During the freezing, flakes form before total solidification takes place. Even after solidification, it tends to break up into flakes very much like pastry. Frozen juice should be analysed soon after melting for deterioration sets in very rapidly.

When kept above freezing, slow hydrolysis takes place. This action commences at 1 degree above zero and at such temperatures as plus 5 it becomes very noticeable after three or four days, especially in infected juices. At 0 degrees, or freezing point which takes place at two or three degrees minus, there is no noticeable action, secondary juices having been kept at the latter temperature for two weeks. Part of this hydrolysis is probably chemical, for biological action is practically nonexistent at such low temperatures, though certain forms of Aspergillus show activity at 1 degree.

Experiments have been carried out at various temperatures, of which results are shown below. There was a tendency to overload the machine in some series, resulting in some little time elapsing before the desired temperature was attained.

Series 1.

In this series, the various juices were collected directly from the mill, polarised and placed in Winchester Quarts in the Electrolux. The overloading was considerable for the size of the machine, and several hours elapsed before they reached a temperature of 5 degrees. At this temperature they were kept for four days, and tested at the end of each twenty-four hours. At this comparatively high temperature there was a considerable drop in polarisation, especially in the case of infected juices. The action was entirely hydrolysis with an increase in invert sugar but no alcoholic fermentation.

	Crusher	Last Mill	Primary	Secondary
Initial			·	Ū
polarisation	71.3	10.3	58.1	18.85
After 24 hours	71.3	8.8	56.1	17.9
,, 48 ,,	70.8	$^{+}7.2$	53.4	17.2
,, 72 ,,	70.4	6.3	50.3	16.4
96	70.1	5.3	47.0	15.3

The results show vividly what happens with slow cooling, and keeping the temperature at too high a degree over a long period of time.

In another experiment, a sample of heavily infected secondary juice was rapidly cooled—in about twenty minutes—to freezing and kept for two weeks.

Initia	l po	larisat	tion	••	 19.8
After	$\mathbf{\tilde{24}}$	hours			 19.8
,,	48	,,		••	 19.9
	144	,,			 20.0
	216			••	 17.6
	288			••	 17.6

Owing to the water and electricity being shut off during the week-end, it was not possible to keep the sample frozen during that period, the temperature rising to plus 8 degrees. The drop in polarisation from 20.0 to 17.6 took place during this period, but was arrested when the sample was refrozen.

Series 2.

In this series, composite samples were made up from the ordinary routine samples. As each hourly sample came in from the factory it was polarised and 25 mls. added to a composite kept in the refrigerator. At the end of twenty-four hours the refrigerator sample was analysed and the polarisation compared to that of the average of the twenty-four hourly polarisations. In the first case the temperature was so regulated that the samples would freeze solid, while in the other cases they were kept at 3 and 1 degree.

Samples Frozen Solid.

	DDIMADA		SECONDARY THICE			
	I KIMARY	JUICE	SECONDARY JUICE			
	Av. 24 hr.	Refrig'tor	Av. 24 hr.	Refrig'tor		
Date	polarisation	sample	polarisation	sample		
7/7/31	56.90	57.0	19.83	19.8		
8/7/31	56.55	56.9	19.92	19.9		
9/7/31	56.90	57.0	20.57	20.6		
10/7/31	56.45	56.5	19.64	19.7		
11/7/31	56.57	56.7	20.45	20.6		
12/7/31	55.85	55.7	19.62	19.8		

The differences noted are all within the limits of experimental error.

Samples maintained at 2 degrees

	bampies i	mannea nica e	it o acgrees.	
14/7/31	55.81	55.9	18.36	18.3
15/7/31	56.00	55.4	19.75	19.6
16/7/31	52.77	52.1	20.59	20.7
17/7/31	56.75	56.0	21.70	21.9
18/7/31	54.13	53.8	19.11	19.4

Samples maintained at 1 degree.

20/7/31	57.17	57.2	22.77	22.6
22/7/31	60.35	60.1	19.48	19.3
23/7/31	59.81	59.4	20.36	20.8
24/7/31	59.00	58.9	19.75	20.0

The differences in this series is so small as to be negligible.

Series 3.

This consisted of a number of complete tests on crusher juice which was stored in the Electrolux for 24 hours. In all cases the juices were brought to freezing point although some little time elapsed before this temperature was reached. Sucrose % Cane determined by assuming a Java Ratio of 75.

INITIAL				24 HOURS						
No.	Ref. Solids	Polar.	Sucrose	Pur.	Suc. % Cane	Ref Solids	Polar.	Sucrose	Pur,	Suc. % Cane
A. B. C. D. E. F.	$19.38 \\ 21.20 \\ 21.90 \\ 21.70 \\ 19.38 \\ 21.08$	57.8 71.7 76.5 74.7 62.8 72.5	$15.33 \\ 18.88 \\ 20.07 \\ 19.63 \\ 16.66 \\ 19.10$	$79.1 \\ 89.1 \\ 91.6 \\ 90.5 \\ 86.0 \\ 90.6$	$11.49\\14.16\\15.05\\14.72\\12.49\\14.32$	$19.21 \\ 21.05 \\ 21.58 \\ 21.93 \\ 19.04 \\ 21.19$	$56.8 \\71.4 \\76.0 \\74.8 \\62.6 \\72.3$	$15.08 \\ 18.82 \\ 19.98 \\ 19.62 \\ 16.63 \\ 19.02$	78.589.492.689.487.389.8	$11.31 \\ 14.11 \\ 14.98 \\ 14.72 \\ 12.47 \\ 14.26$

The small drop in polarisation was due to the slow cooling of the samples owing to their bulk.

Further tests on juices all showed that when frozen solid they would keep in a perfect state of preservation.

Tests on bagasse were less successful owing to the difficulty of cooling down the large quantity required for the sucrose test. Although a considerable drop in sucrose was recorded, there was very little change in moisture content of a sample placed in a loosely covered container after twenty-four hours. Another sample of bagasse in an unstoppered Erlenmeyer flask showed a negligible loss of moisture after three weeks storage.

From these tests we have reached the conclusion that if the samples be brought to freezing point rapidly—say fifteen or twenty minutes—refrigeration offers an ideal system for preservation of cane juice samples, even though they be heavily infected.

In a modern factory of large capacity the number of uice samples that have to be tested hourly are such as to demand the major portion of the shift chemist's time and as the capacities of the plants are increased, it is becoming increasingly difficult to cope with the volume of work presented. For this reason it seems to us desireable to sub-sample the juice, and accurately analyse the composite samples every eight or even twelve hours, thus relieving the shift chemist of much unnecessary work to the benefit of a closer supervision of the problems arising from time to time; and at the same time assuring a more accurate control, and greater all round efficiency of the factory.

Mr. FOSTER continued: We further put this refrigeration to more practical purposes. Where one of the Planter's chemists was in some doubt as to the purity of the juice, we told him to place the samples in the refrigerator, and it would be tested in the morning. In addition, we showed that it was quite practicable to freeze a juice solid, pack it well, and it could then be taken to the Experiment Station for purpose of checking up should any Planter so desire his test to ge checked. Dr. Hedley will remember we took some juice in a solid state to the Experiment Station. We did not try the refrigerator with filter press cake, as it was not suitable for cooling down such a bulk in a hurry.

CHAIRMAN: One point of interest in this is that it is breaking new ground. It is one which will probably present to your minds several queries. There is one thing which appears to me from the figures. In the case of samples frozen solid there appears to be a slight tendency for the polarisation to increase. The changes are only perhaps within the limits of experimental error, but there seems to be a general tendency in that direction. Take the second lot of figures on the second page, five out of six of the refrigerator samples were slightly higher than the original sample. It seems probable that these juices in freezing do not freeze particularly homogeneously-in fact, it would be strange if they did-and doubtless certain parts of the water freeze out before the solids freeze, and when handling the frozen mass afterwards it seems to me there is a danger of losing some of the water; some of it may be allowed to drip off, and your subsequent brix will be higher than the original. I don't know whether Mr. Foster has found that out.

Mr. FOSTER : In the case of the first set of figures, the secondary juice, that was a very badly infected sample-one which generally drops about three to four degrees in the course of half an hour. That was put into the refrigerator and re-polarised after 24 hours, and as the refrigerator was blank that night we thought we would put it back to see how the pol went. In the subsequent pol the sample was not melted thoroughly, there were still a few solid flecks, therefore it is quite probable the objection you raise is the explanation for this rise in pol which was noted from 19.8 to 20. With regard to the other juice frozen, with results observed after 24 hours, I think that was only experimental error. In further work we obtained close readings and small discrepancies all within the limits of experimental error.

CHAIRMAN: I have been asked by Mr. Watson to enquire how long it takes after removing the sample from the refrigerator to get it ready for testing. What temperature is it polarised at?

Mr. FOSTER: What we worked out eventually was to withdraw the sample from the refrigerator when frozen. We generally stored it in an Erlenmeyer flask and placed it in hot water to melt it rapidly, and bring the temperature to 20 degrees, which was generally in about ten minutes. The analysis was then proceeded with quickly, so as not to give the sample any chance to deteriorate after we had melted it. Some of the experiments we conducted showed juices after storage for a long period did go off very quickly indeed, hence the need for haste after melting. With a juice frozen solid, there is no reason why you cannot carry out all your pol at the temperature mentioned. It is only a matter of practice to get the required degree of cooling.

Dr. HEDLEY: I do remember Mr. Foster coming along to the Experiment Station one day. He brought along a "billy can," and he called me quietly outside and said, "Come and see what I have got." It was a hot day, and I said, "What have you got there?" He said, "Something in ice." Of course, my mouth began to water ! (Laughter.) He opened it, but it was nothing but ice and sugar-quite unsatisfactory for all that mystery ! But I do think you will agree with me that Mr. Foster has got a really splendid idea. Can you imagine anything more glorious on these hot summer nights than to have a refrigerator in your lab.! (Laughter.) But from another practical point of view, I think that these experiments should be carried on. It is probably a thing that we might bring to the Millers' notice and have it tried out in several factories. If we do not do something like that the thing will be allowed to drop, as so many of these things have been allowed to drop; and if, as it appears to be the case, it will lead to less work in the laboratory, it is very worthy of consideration and encouragement. The sample which came to the Experiment Station was certainly in a solid state, and in the event of a dispute-which I trust never occurs in the mills !--we would have the Experiment Station as an unbiased body to decide such a dispute. There is only one thing I might mention : Mr. Foster should have used a thermos flask. If you can transport liquid air in such a flask, then frozen sugar liquid can be conveyed.

Mr. RAULT: I would like to know from Mr. Foster whether, when dealing with refrigerated juices, he has noted any peculiarity in the brix determination. On submitting raw juice to heat, its physical nature is altered through coagulation of certain suspensoids which are brought down and produce partial clarification. One could expect that freezing raw juice would likewise produce some change in its physical constituents. How far these changes may affect the brix reading it is hard to say, but there is a possibility of altering the brix reading before and after cooling, and accordingly influencing the purity test, which is of great importance in the system of bonus and penalties as laid down by the Fahev agreement.

Mr. FOSTER: In those tests shown in Series 3 we have not taken the brix, as we could not cool off sufficiently quickly. However, we have taken the refractometer results. The refractometer we used was not in the best condition, and probably 0.1 is about the limit of its accuracy. The division line is not absolutely clear with the raw juice. You will notice, however, that the brix as returned by the refractometer we did use did not show any very material difference on the tests shown. Certainly, in the case of sample "C," we have a difference of 0.3; "D," on the other hand, is actually higher. "E" is lower, and "F" is practically the same. Therefore, I do not think there is any difference in the brix of the sample after testing. However, further investigations could be carried out on that point. Probably, if a machine could be obtained which would freeze a large sample rapidly, it would be possible to test your brix; but with the ordinary refrigerator such a thing is not possible-you could only freeze a small quantity.